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TECHNICAL NOTE 3055

FRICITION AND WEAR INVESTIGATION OF

MOLYBDENUM DISULFIDE

I - EFFECT OF MOISTURE

By Marshall B. Peterson and Robert L. Johnson

Lewis Flight Propulsion Laboratory
Cleveland, Ohio



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SUMMARY

Molybdenum disulfide MoS_2 is generally a very effective solid lubricant. However, very little information is available as to the role of moisture, shear area, surface finish, and several other variables on lubricating effectiveness of MoS_2 . Studies were therefore conducted with a low-speed kinetic-friction apparatus to clarify the role of these variables.

Coefficients of friction were greater at high humidity than with dry air. MoS_2 powder (not prebonded) did not adhere to steel surfaces at high humidity and, as a result, metallic contact was greater and friction increased. Coefficients of friction also increased with higher humidity with thick bonded films where metallic contact was improbable. For the case of lubrication by means of unbonded MoS_2 powder, wear increased as humidity was increased, and increased metallic contact and corrosion occurred. Steel specimens were corroded by acids formed on contact of moisture with MoS_2 . Variations in shear area resulted from changes in humidity, slider geometry, surface finish, and method of film application. Larger shear areas caused greater friction when MoS_2 filled the surface interstices and was sheared.

INTRODUCTION

Considerable research has been reported (refs. 1 to 5) showing that MoS_2 has good properties as a solid lubricant. The effectiveness of MoS_2 , which has been accepted as a lubricant in many fields and by the military services, is believed to result from its capacity to form a low-shear-strength film on metallic surfaces, thus reducing both friction and wear. The literature contains many references to the coefficients of friction obtained when MoS_2 is used in greases, oils, water, bonded films, sintered metals, and as a dry powder (for

example, refs. 3 and 5). Very little basic information is available, however, as to the effect of such variables as moisture, contact area, and surface finish. It has been established that moisture can be either beneficial or harmful in lubrication depending on the type of lubricant. For example, moisture is beneficial in lubrication by graphite and, as shown in references 6 and 7, the presence of adsorbed gas or water in graphite was essential for effective lubrication. On the other hand, Campbell has found that with steel surfaces adsorbed moisture will increase the coefficient of static friction for both unlubricated surfaces (ref. 8) and surfaces boundary lubricated with commonly used types of fluid lubricant (ref. 9).

Knowledge of the effect of moisture on lubrication by MoS_2 is important because, in general practice, it will be used under widely varied moisture conditions resulting from climatic changes. The use of water mixtures is also important, since that is a common method of applying solid lubricants. Therefore, the research reported herein was conducted to investigate the effect of moisture on the lubricating effectiveness of MoS_2 , which is defined herein as the ability of the lubricant under specific conditions to maintain low friction and to prevent surface failure and high wear.

Accordingly, a series of experiments was conducted at the NACA Lewis laboratory primarily to determine the effect of moisture on friction and wear of steel surfaces lubricated by MoS_2 ; in addition, the secondary effects of shear area, running time, and surface finish on the lubricating properties of MoS_2 were considered.

APPARATUS AND PROCEDURE

Test Specimens

A 2-inch-diameter ring specimen rotated on edge against the flat surface of a disk specimen (fig. 1). The ring shown in figure 1 is a test cup ordinarily used as a test specimen in the SAE lubricant test machine. The ring is made of nickel-molybdenum steel (SAE 4620) case hardened to Rockwell C-62. Three nodes were ground on the edge of the test ring that rotated against the disk surface. Each node was in the shape of a cylinder (2 in. radius), and line contact theoretically existed between the three sliding nodes and the disk. The nodes were ground on the rings using a jig and a master pattern machined to close tolerances; therefore, each ring ground was essentially identical with all others. Final grinding marks were removed by hand polishing.

The disks were machined from SAE 1020 steel. Unless otherwise specified, they were given a vapor-blast finish to avoid the directional nature of ground surfaces. The surface roughness of these specimens was 60 rms as measured with a profilometer.

Apparatus

The apparatus used in this investigation is shown in figures 2 and 3. The ring specimen was mounted in a holder which was held in the spindle of a rotating shaft as shown in figure 3. The rotating shaft could be raised or lowered by means of a rack in the rear of the spindle and a pinion gear in the support frame; it could also be clamped in any position. The spindle was rotated at 11 rpm (5.7 ft/min at a radius of 1 in.).

As shown in figures 2 and 3, the disk specimen of figure 1 was mounted on a support table. A pin on the surface of the support table meshed with a hole in the disk specimen and prevented relative motion between the two. The support table was mounted on three springs which compensated for horizontal misalignment and ensured approximately equal distribution of load among the three nodes of the ring specimen. The bolts served as spring guides and transmitted frictional torque to the lower assembly. Results obtained with this apparatus have satisfactorily checked accepted coefficients of friction for various dry metal combinations and specific boundary lubricants.

A load was applied between the specimens by lowering the rotating assembly to deflect the base springs (fig. 2). The magnitude of the load was obtained by observing the amount of deflection as indicated by a dial gage. A calibration curve of deflection versus load was obtained with dead weights. During a test run the desired load was first applied and the spindle then clamped in that position, thus maintaining the load constant throughout the test.

Frictional torque was measured by restraining the motion of the lower assembly (which supported the flat disk) through a dynamometer ring on which strain gages had been mounted. The deformation of the dynamometer ring was indicated by a calibrated potentiometer. A ball bearing at the base of the lower assembly allowed the lower assembly to rotate freely with negligible friction.

A chamber (shown in fig. 2) rested on the support table and enclosed the test specimens. Air of controlled humidity could be maintained in the vicinity of the test specimens.

Cleaning Procedure

Prior to each test run, both of the friction specimens were cleaned according to the following procedure:

- (1) Washed in 50:50 acetone-benzene solution
- (2) Scrubbed with repeated applications of moist levigated alumina
- (3) Washed in tap water
- (4) Washed in distilled water
- (5) Washed in 95-percent alcohol
- (6) Dried and stored in a desiccator

Method of Varying Air Humidity

For this investigation, dry air was obtained from a -70° F refrigerated air line, filtered through a column of glass wool, and heated to room temperature. After passing through the glass wool, part of the air passed directly to a mixing flask and part of it bubbled through distilled water and then passed into the mixing flask (fig. 2). From the mixing flask, the air passed into a flask for measurement of relative humidity by means of a hair hygrometer. (The hygrometer was calibrated and frequently rechecked with a sling psychrometer and a dew-point potentiometer. From 6 to 65 percent relative humidity, the values are believed to be accurate to within ± 2 percent of the reading; outside of this range of humidities, the accuracy of this hygrometer was poor.) From this flask, air passed into the test-specimen chamber at a rate such that a slight positive pressure existed within the chamber. The relative humidity could be varied by changing the relative amounts of air which passed directly to the mixing flask and that which was bubbled first through water. The rate of air flow and the room temperature were held constant throughout the experiments.

In experiments to determine the effect of shear area at a time when the refrigerated air system was inoperative, air from the laboratory compressed air system was dried by bubbling through sulfuric acid and then filtered through glass wool.

Test Procedure

The test specimens were cleaned according to the previously mentioned procedure, placed in the test chamber, and dried for $1\frac{1}{2}$ hour

by allowing dry air to flow through the enclosing chamber. The chamber was then opened and enough pure MoS_2 was spread over the surface of the disk to cover it to a depth of $1/4$ inch (approx. 20 g). The MoS_2 was then dried for $1/2$ hour. After this drying period, the humidity was adjusted to the desired level; after an additional $1/2$ hour with air at controlled humidity passing through the test chamber, the run was begun. The tests were of 6-hours duration and were made with a constant load of 40 pounds and at a constant speed of 11 rpm, which corresponds to a sliding velocity of 5.7 feet per minute. Friction force was continually observed and measurements were recorded approximately every 10 minutes. At the end of each run, the test specimens were studied with a microscope and photographed and the wear area of the slider obtained by use of a planimeter on an enlarged image.

RESULTS

Effect of Humidity

Friction data are shown in figures 4(a) and (b) as a function of time with humidity as a parameter; and in figure 4(c) a cross plot shows directly the effect of humidity on coefficient of friction at various times during the runs. The lowest and most stable friction values were obtained at the minimum humidity (figs. 4(a) and (c)). Friction coefficients were increasingly higher at higher relative-humidity levels up to between 55 and 65 percent. At low humidities (fig. 4(a)), friction curves were easily reproduced. Erratic friction values were obtained at high humidities (fig. 4(b)); however, friction was decreased very markedly by increasing the relative humidity above 65 percent. There was little effect of running time on friction at the minimum humidity level (fig. 4(a)), but an increasing influence of running time on friction was observed as the relative humidity levels were increased (fig. 4(b)). The data of figure 5 show that wear was greater as the relative humidity levels were increased. At the highest humidities, wear increased with humidity although friction was decreasing as humidity increased.

Photographs of the track and one node of the slider were taken at the conclusion of all the test runs; several examples are shown in figure 6. In general, the MoS_2 films in the photographs have a light gray matte appearance; metallic surface asperities generally show greater reflectivity and the black spots are loose particles of MoS_2 . Microscopic observations of the contacting areas show that at low humidities (6 and 15 percent) the MoS_2 adhered to the slider and the disk as illustrated in figure 6(a) and continual shear of the MoS_2 took place; the films were continuous and smooth. In the

31, 44, and 65 percent runs (as shown in figs. 6(b) and (c) for the 44 and 65 percent runs), the film on the disk was smooth but in many places the surface asperities could be seen to protrude through the film. At the higher humidities, the film on the disk became disrupted and non-continuous with large voids apparent in the lubricant film while on the slider only slight traces of MoS_2 remained (fig. 6(d)). The MoS_2 came off the slider at lower humidities than those at which the disk film became disrupted. After runs at high humidities, the metallic surfaces were slightly stained as if etched where they had been contacted by the MoS_2 .

Effect of Shear Area

In experiments to determine the effect of shear area on friction force, the shear area was varied by two methods: (1) A noncontinuous film was formed by using an amount of MoS_2 powder that was insufficient to fill the interstices, thus restricting the shear area to the tips of the metallic asperities (as shown in fig. 7); this was accomplished by lightly wiping MoS_2 on the surface using lens tissue. (2) The nodes of the slider specimens were flattened by abrading to obtain various apparent contact areas and were run using the usual excess amount of MoS_2 powder which filled the interstices and gave a continuous film.

The friction values obtained with disks having noncontinuous wiped films at approximate relative humidities of 6 and 65 percent are compared in figure 8 with those of the disks that initially had continuous films (humidities, ≤ 6 and 55 to 65 percent) of figure 4. It is apparent that when the shear area is restricted (noncontinuous as in fig. 7) a considerable reduction in the friction coefficient is obtained at both high and low humidity (as shown in fig. 8).

When different areas were ground on the slider nodes and these run against continuous films of MoS_2 on the disk at less than 6 percent relative humidity, a similar decrease in friction coefficient was observed (fig. 9) with a decrease in shear area. Shear area was considered as the area of MoS_2 visible on the slider over which contact had occurred as measured with a planimeter on a projected image. This decrease in friction force with a decrease in slider shear area was also observed at higher humidity, but quantitative results were difficult to obtain, since the actual shear area was not as well defined.

Effect of Surface Finish

There was a large dependence of coefficient of friction upon the surface finish of the disk. For example, when a ground disk specimen was used in place of the vapor-blasted specimen, the friction force during one revolution of the slider fluctuated over a large range of values (0.1 to 0.25). The lowest value was obtained when the nodes were sliding approximately parallel to the grinding marks, the highest value when sliding perpendicular to the grinding marks. Friction was high when build-up of large shear area occurred by the trapping of MoS_2 in the grinding marks when sliding perpendicular to these marks; friction was lowest when sliding parallel to the marks and the shear area was limited to the area of the tips of the grinding marks. Various surface finishes were observed to influence plastic flow of MoS_2 in different ways, causing different shear areas and yielding corresponding friction coefficients.

Effect of Adding Water to MoS_2

Further data on moisture effects were obtained by making runs with either 5, 10, or 50 percent (by weight) water added to the MoS_2 powder before it was spread on the disk. Additional runs were made for comparison using only pure distilled water applied to the disk surface. These data are shown in figure 10. Low initial values of coefficient of friction were observed in the 5- and 10-percent-water runs; with pure water the initial value was high. A photograph of the disk surface of a 10-percent run after 5 minutes sliding is shown in figure 11; it can be seen that the MoS_2 (light areas) has adhered to the surface of the disk in relatively few places at the tips of the asperities and thus the shear area was low. With continued running time, the friction with 5 and 10 percent water present increased to a higher level (fig. 10). This level of friction values is probably associated with increased metallic contact. The friction trends for minor water additions may be affected by continual evaporation of moisture from the powder. In no instance did the friction values for mixtures of MoS_2 in water become as high as the comparative data for pure water.

During the experiments with mixtures of water and MoS_2 reported herein, it was observed, as has been reported by others (ref. 10), that water became acidic upon contact with MoS_2 . As previously mentioned, after friction runs at high humidity and also with mixtures of water and MoS_2 , steel surfaces were stained as if etched by acids.

Effect of Humidity with Bonded Films

Bonded films of MoS_2 may have more practical usefulness than loose powder. Consequently, experiments were run to determine if humidity had a similar effect on MoS_2 films bonded to the surface. In one experiment the film was bonded to the disk specimen only, and in a second experiment bonded to both the disk and the slider. These specimens were run for 30 minutes at the lowest humidity obtainable (less than 6 percent); after 30 minutes running time, the air was bubbled through water which increased the humidity to approximately 85 percent. An appreciable increase in friction coefficient was observed (fig. 12) as the humidity was increased. In general, this effect was reversible with friction coefficient decreasing again when dried air was admitted. Similar runs with powdered MoS_2 showed the same trend.

The friction coefficients and the shear areas were higher for cases where bonded films were applied to both specimens than when only one surface had a bonded film. In one comparison the measured shear area with both specimens having bonded films was three times greater than when only one specimen had a bonded film.

DISCUSSION

Bridgman (ref. 11) has reported that solids similar to MoS_2 flow plastically during shear. The observed friction data can be partly explained by assuming that in the experiments reported herein the MoS_2 behaved as a plastic material and not according to established friction concepts for clean metals or for conventional fluid boundary lubricants. The theory for friction behavior by thin solid films as developed by Bowden and coworkers (ref. 4) is believed to be generally adaptable. Plastic flow by MoS_2 under shear would fill the surface interstices (if sufficient MoS_2 was available) and, therefore, the area over which shear takes place during sliding will not necessarily be limited to the "real area" in contact (in the discussion of data reported herein "real area" means the contact area that would be obtained with unlubricated base metals).

Friction force depends on the strengths of the materials sheared and on the shear areas. Although some metallic contact may have occurred that was not detectable with a microscope, it appeared that only MoS_2 was sheared at low humidities. As humidity levels were increased, greater numbers of metallic asperities became visible, because moisture prevented MoS_2 from adhering on the surface areas of load-carrying asperities; friction force also increased, because the exposed

metal contacts had greater shear strength than the MoS_2 . Thus, the total shearing force (or effective shear strength), but not necessarily the total shear area, was increased.

The double-bonded films were approximately 1/16 inch thick after running. In that case, it was unlikely that metallic contact occurred; therefore, the friction data indicate that effective shear strength of a bonded MoS_2 film alone can increase with increase in humidity. Shear strength of MoS_2 is also decreased by orientation (ref. 5), as is graphite (ref. 9). The adsorption of moisture on the crystal surfaces of graphite decreases its shear strength (refs. 6 and 9); but MoS_2 does not exhibit similar behavior. The reason for this lack of similarity has not yet been established.

Reference 2 indicates that shear strength of MoS_2 varies slightly with pressure. In the present study the changes in pressure were small and hence introduced negligible changes in the results.

The area over which shear takes place can also vary in several ways. Bowden (ref. 4, p. 114) has shown that with plated metallic films, the film thickness determines the area of shear and thus the coefficient of friction. In experiments with powdered MoS_2 , several factors were observed to effect shear area and probably also affected the film thickness. These factors are moisture, slider geometry, method of film application, and surface finish. The initial presence of excessive amounts of MoS_2 caused the formation, at low humidities, of films that provided effective lubrication for much greater periods of time than the 6 hours of the experiments reported herein. With dusted films, which gave very low coefficients of friction, it would be necessary to provide a means of supplying additional MoS_2 in order to obtain continuous effective lubrication.

It is speculated that moisture had its adverse effects on lubrication by MoS_2 , because it became adsorbed on the metal surfaces and, among other things, prevented the MoS_2 from forming an adherent film. As mentioned, with runs at 31 percent and more humidity, load-carrying metallic surface asperities were visible; the number and area of such asperities increased with humidity. When more moisture was available (above 70 percent relative humidity and with water additions), bulk MoS_2 was prevented from adhering in surface interstices, leaving voids in the shear area.

The reasons for the reduction in friction with humidity above 70 percent are not understood. The reduction in shear area from the

formation of voids in the MoS_2 film would tend to reduce friction; however, the higher humidity would also expose more metallic contacts, which would tend to increase friction. The formation of low-shear-strength corrosion products on the metallic asperities would tend to decrease friction; however, in runs with water alone which had been made acidic by previous contact with MoS_2 , there was no substantial decrease in friction below that obtained with pure water.

The increase in wear with greater availability of moisture could be explained on the basis of either increased metallic contact or more corrosion. Corrosion is a form of wear, and study of the specimens indicated that corrosive etching, in general, increased as did wear with greater moisture availability. The corroding acids formed on contact of water with MoS_2 may be the product of oxidation of MoS_2 or of residual materials from commercial processes.

These data indicate that lubrication of steel surfaces with MoS_2 is influenced by many factors which contribute to the effective shear strength and the area over which shear occurs. The adverse influence of moisture indicates that the most effective use of MoS_2 would be at low atmospheric pressure, high temperatures, or at other conditions where little or no moisture is present.

CONCLUSIONS

An experimental study of the lubricating properties of molybdenum disulfide was conducted at room temperature to show the effects of moisture and shear area on friction and wear. The following results were obtained at a constant load (40 lb) and at a constant speed of 11 rpm (sliding velocity of 5.7 ft/min):

1. Coefficients of friction were greater at high humidities than with dry air. Molybdenum disulfide powder (not prebonded) did not adhere to steel surfaces at high humidities. Consequently, as humidity increased more metal surface asperities were exposed and coefficient of friction also increased. Humidity may also have increased the shear strength of a bonded film, since friction increased with high humidity when film thickness was so great as to make metallic contact improbable.
2. For the case of lubrication by unbonded MoS_2 powder, wear increased as humidity was increased. This relation could result from increased abrasion between unlubricated metallic asperities; the number and area of such asperities increased with humidity. Corrosion of steel specimens by acids formed on contact of moisture with MoS_2 where the moisture was provided by the humid air increased as humidity was increased and may account for part of the increased wear.

3. Coefficients of friction increased as shear area became larger (the shear area includes the combined areas of metallic contact and of MoS_2 film sheared). Shear area varied with changes in humidity, slider geometry, surface finish, and method of film application. When sufficient material was available, MoS_2 filled the surface interstices and was sheared during sliding. In that case, the total shear area was much greater than the area of contact that would be obtained with unlubricated base metals.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, September 24, 1953

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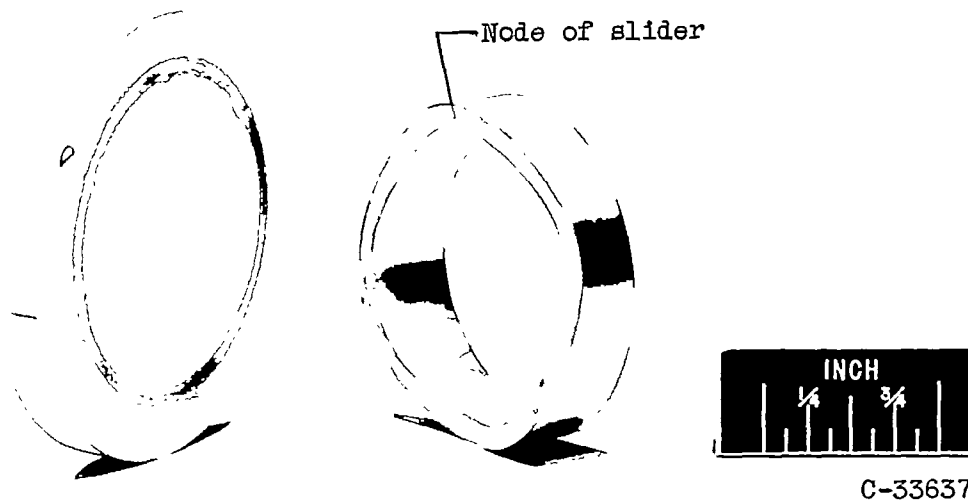


Figure 1. - Friction specimens.

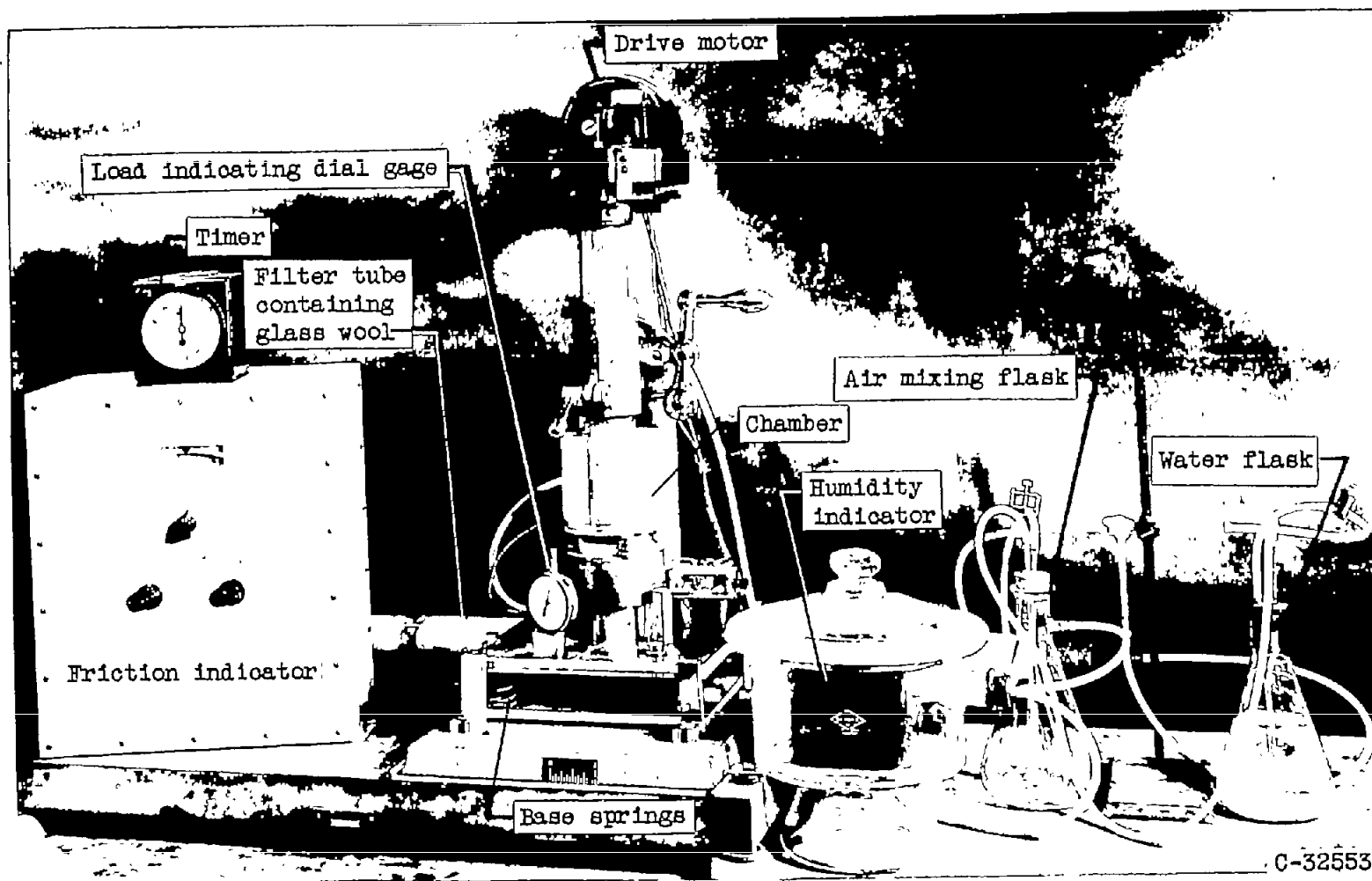
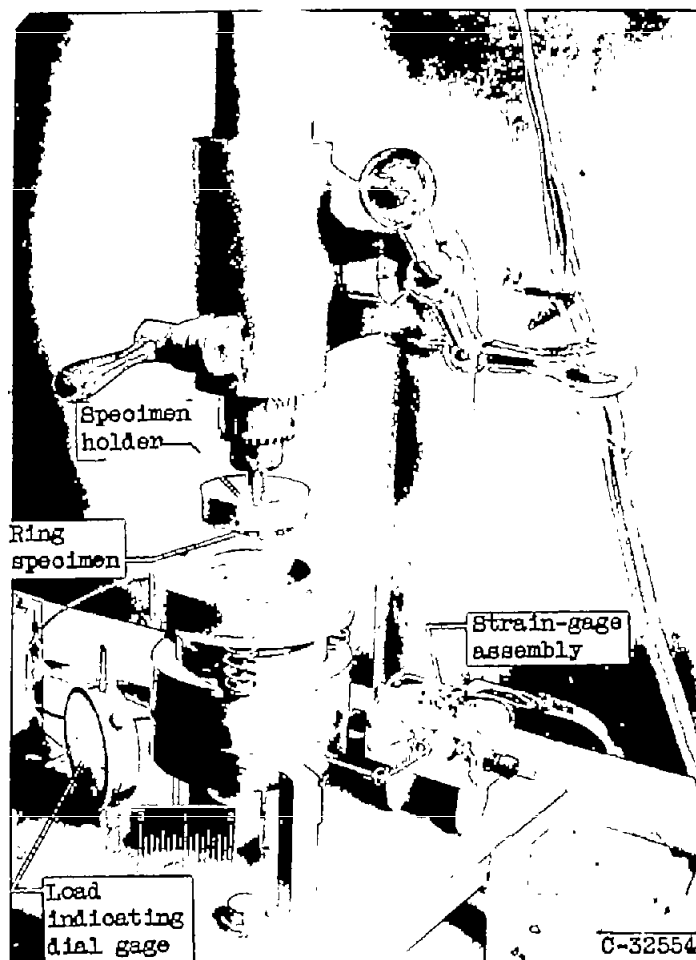
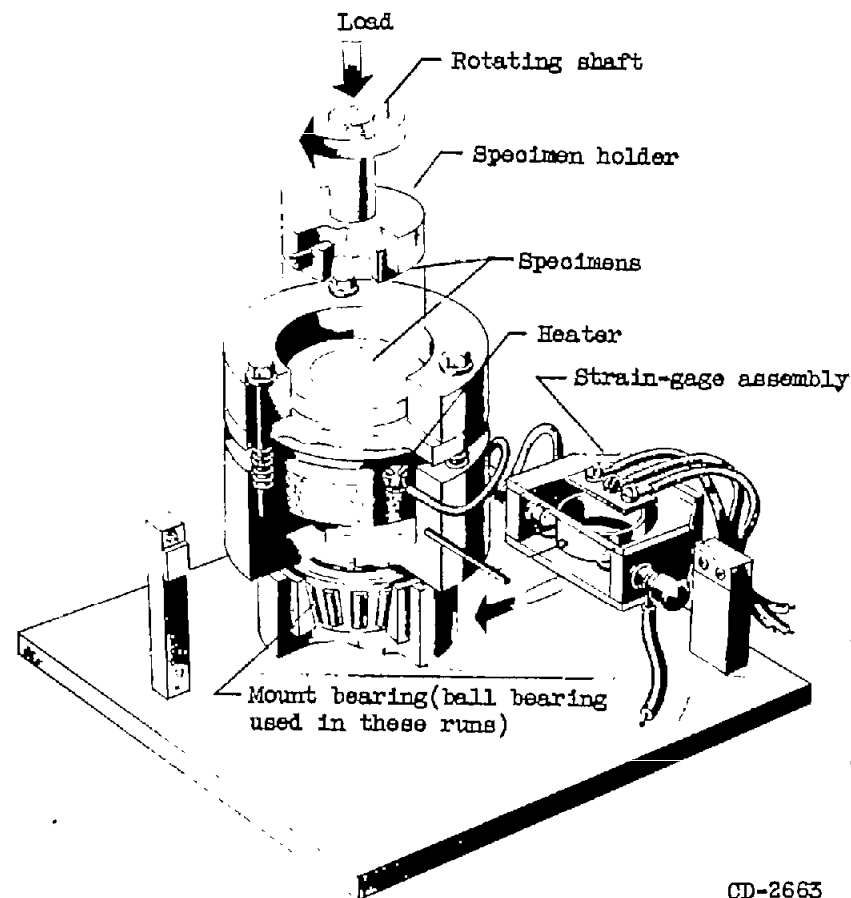


Figure 2. - Kinetic-friction apparatus.



(a) Photograph.



(b) Diagrammatic sketch.

Figure 3. - Detail of basic elements of kinetic-friction apparatus.

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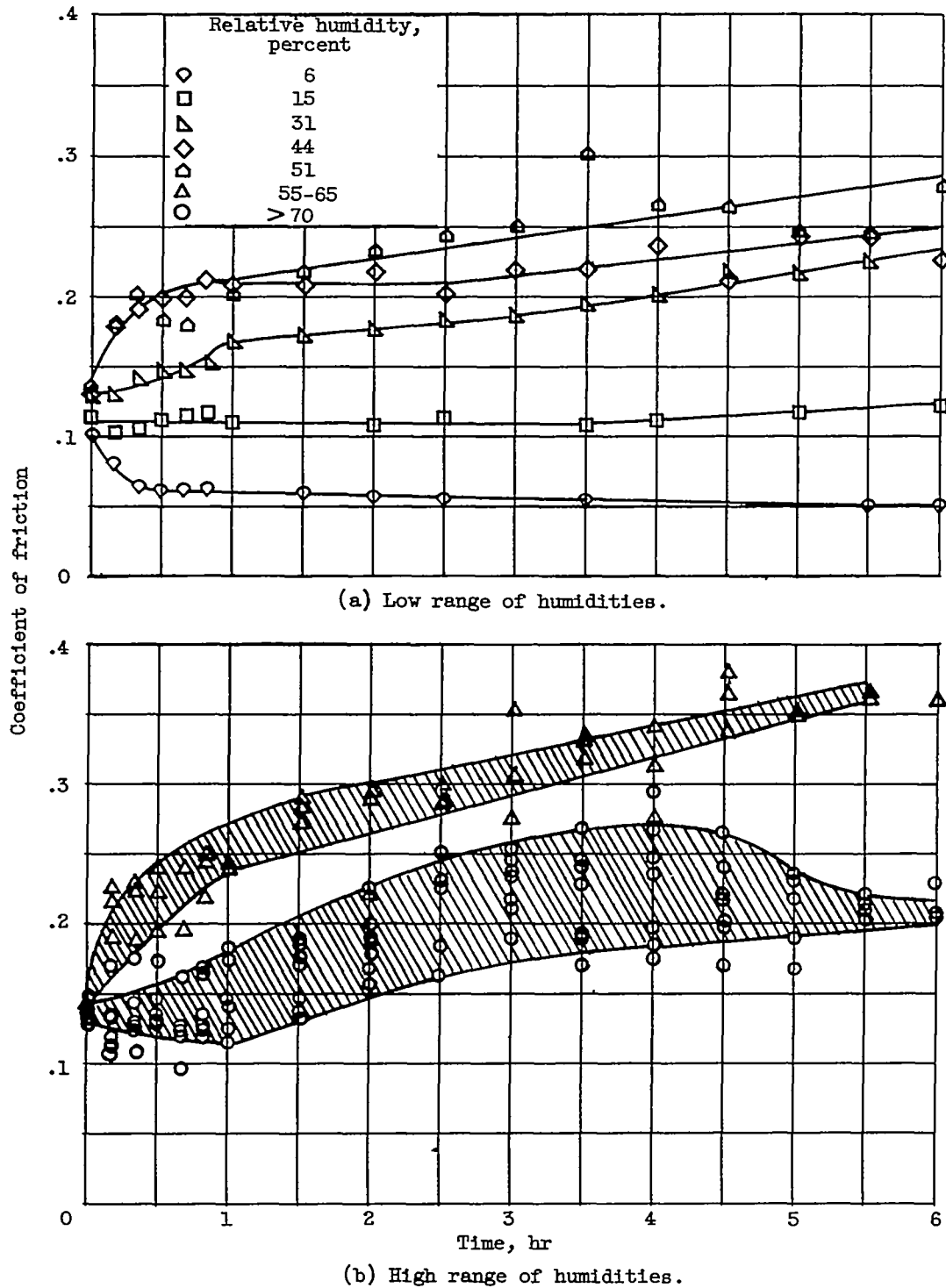
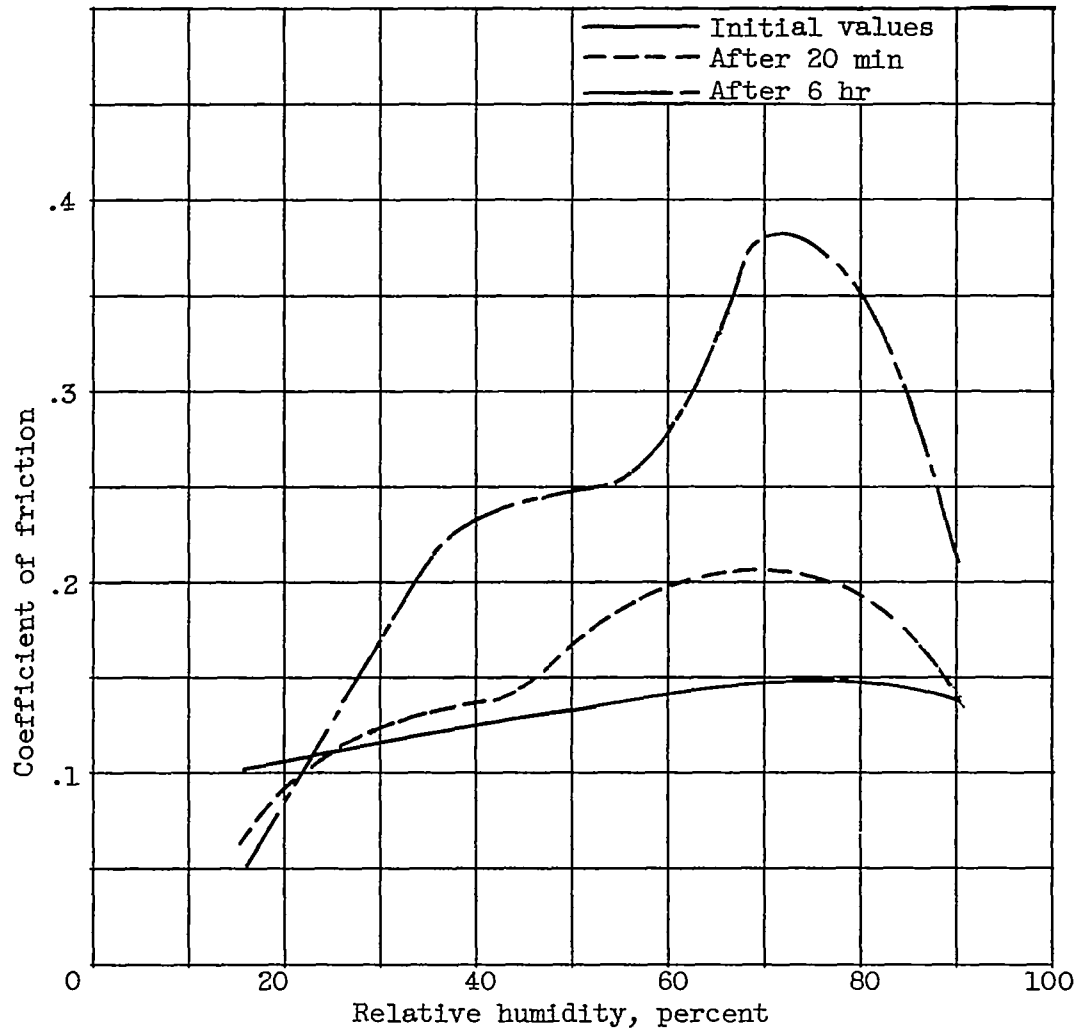


Figure 4. - Effect of various relative humidities on the coefficient of friction of steel specimens lubricated with powdered MoS_2 . Load, 40 pounds; speed, 11 rpm (5.7 ft/min).



(c) Cross plot of data from figures 4(a) and (b)
(data from fig. 4(b) are average values)

Figure 4. - Concluded. Effect of various relative humidities on the coefficient of friction of steel specimens lubricated with powdered MoS_2 . Load, 40 pounds; speed, 11 rpm (5.7 ft/min).

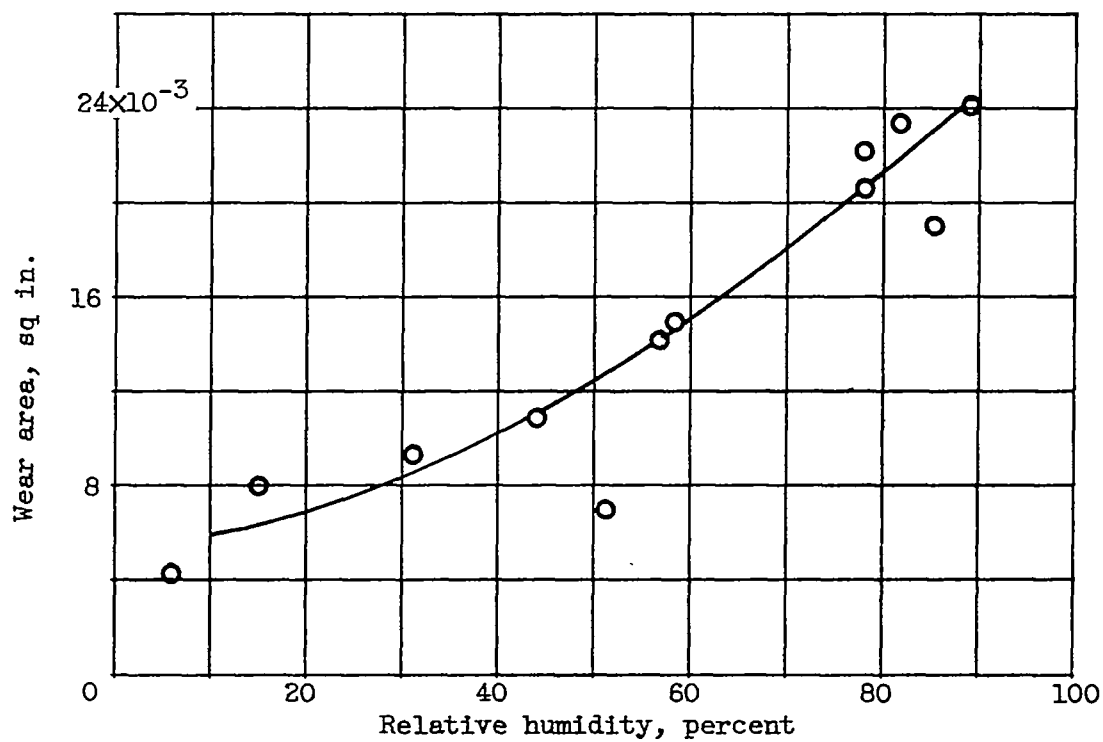
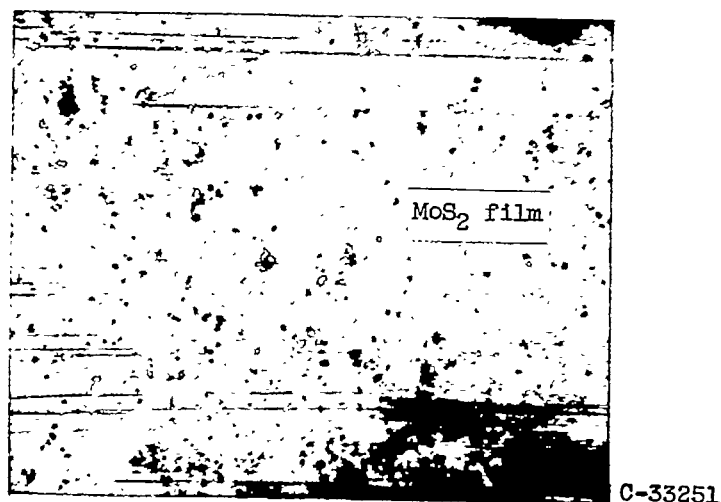


Figure 5. - Effect of relative humidity on slider wear area after 6 hours sliding time; steel specimens lubricated with powdered MoS_2 . Load, 40 pounds; speed, 11 rpm (5.7 ft/min).



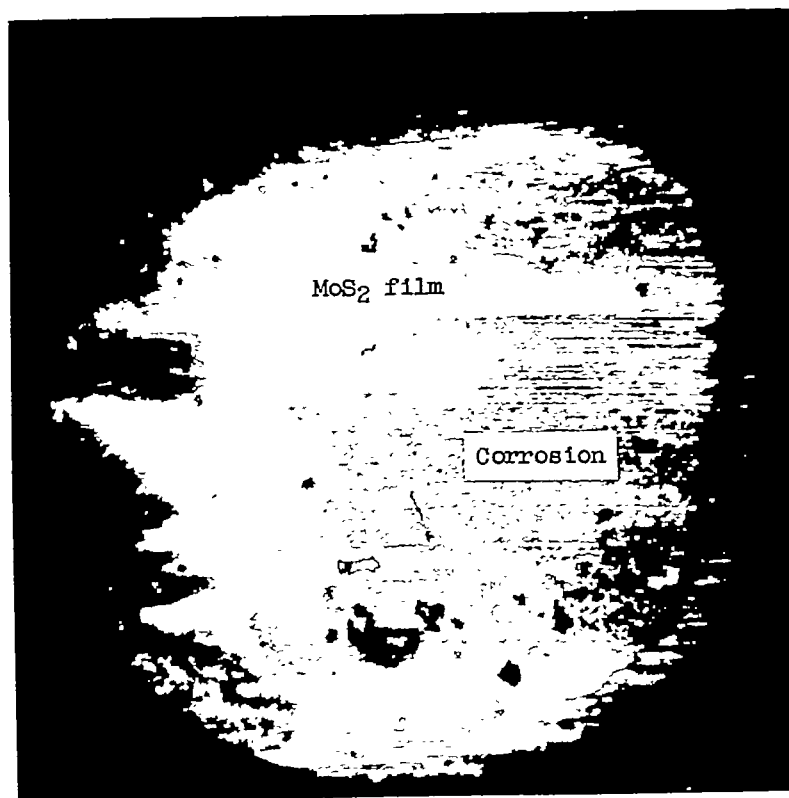
Node of slider



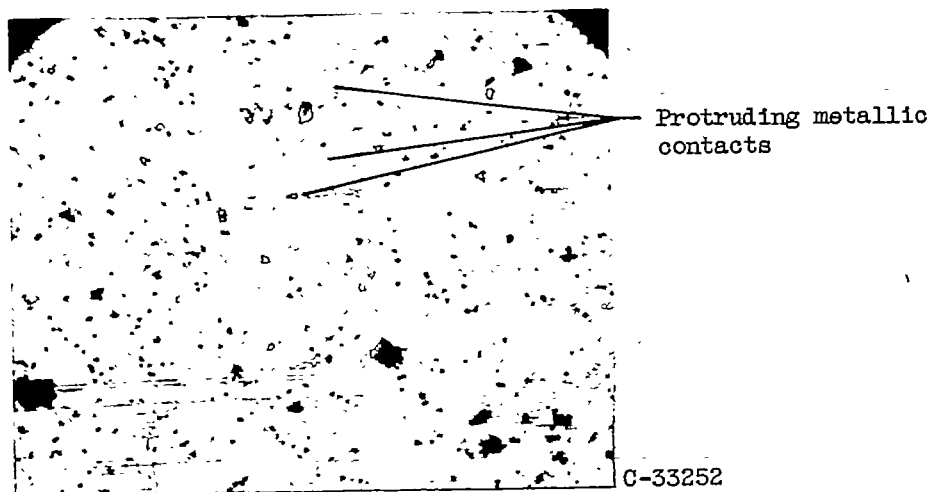
Disk

(a) Relative humidity, 15 percent.

Figure 6. - Photomicrographs showing surfaces of disk and slider after 6 hours sliding time at various humidities. Steel on steel lubricated with powdered MoS₂. Magnification: disk, X100; slider, X50.



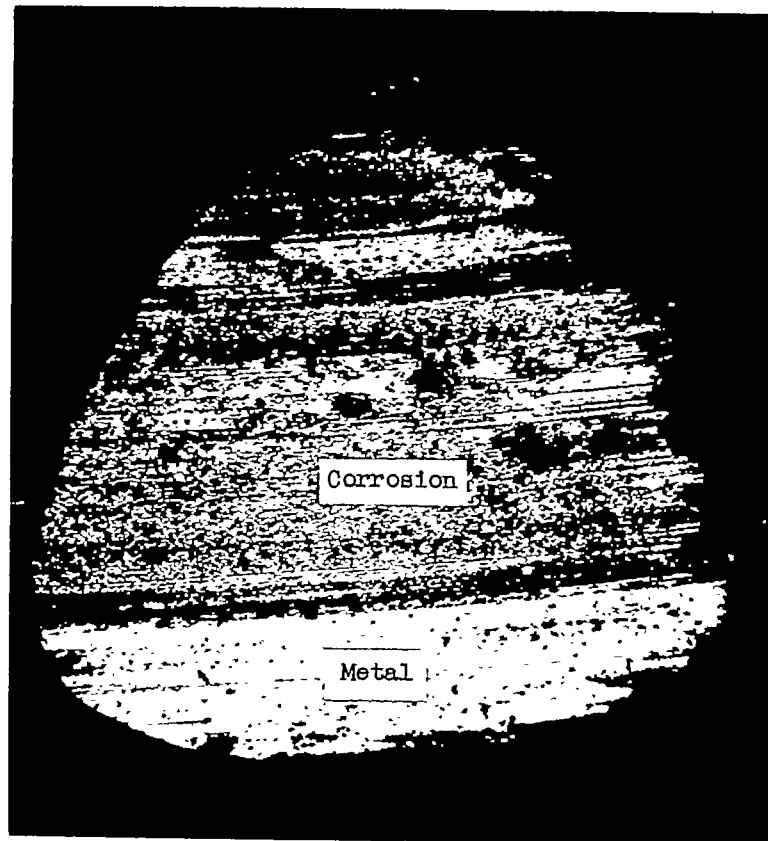
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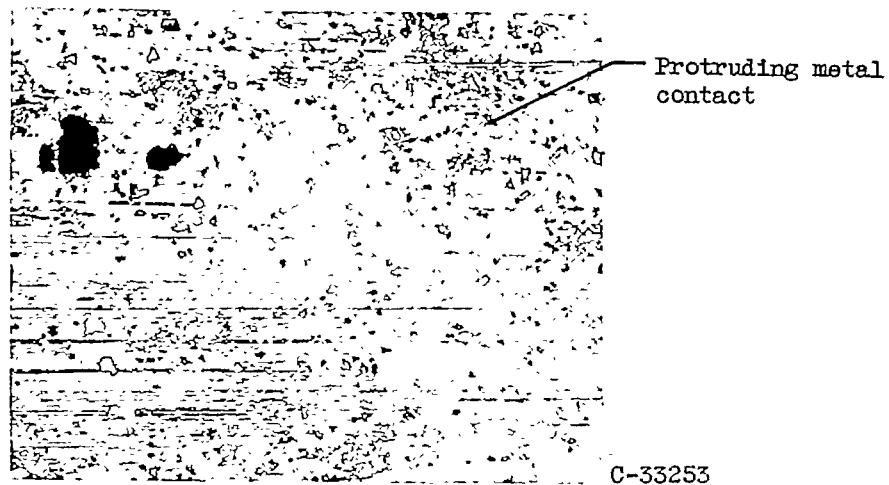
Disk

(b) Relative humidity, 44 percent

Figure 6. - Continued. Photomicrographs showing surfaces of disk and slider after 6 hours sliding time at various humidities. Steel on steel lubricated with powdered MoS₂. Magnification: disk, X100; slider, X50.



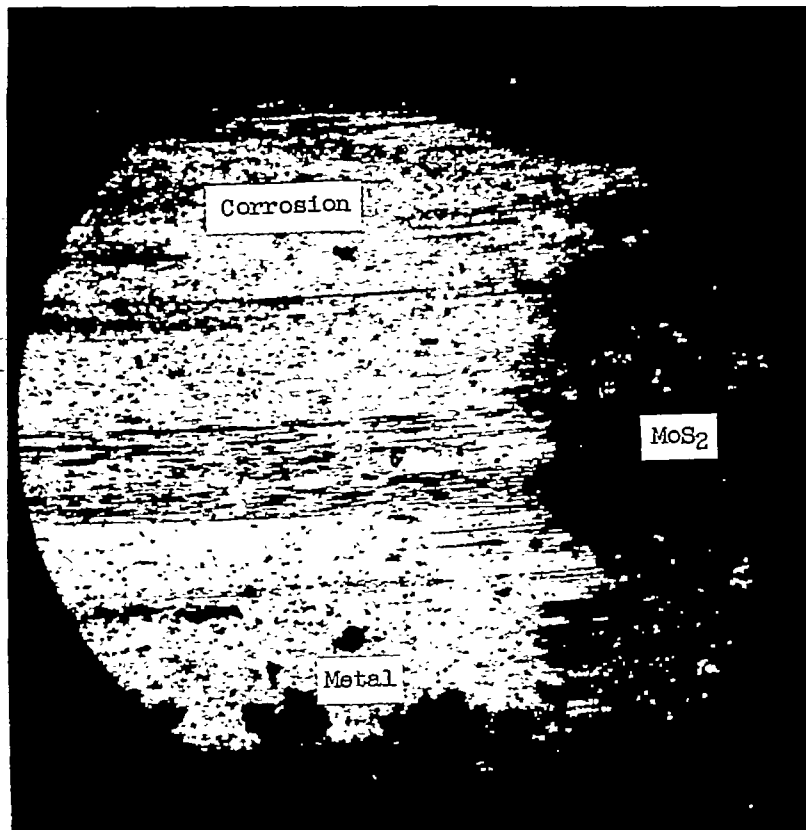
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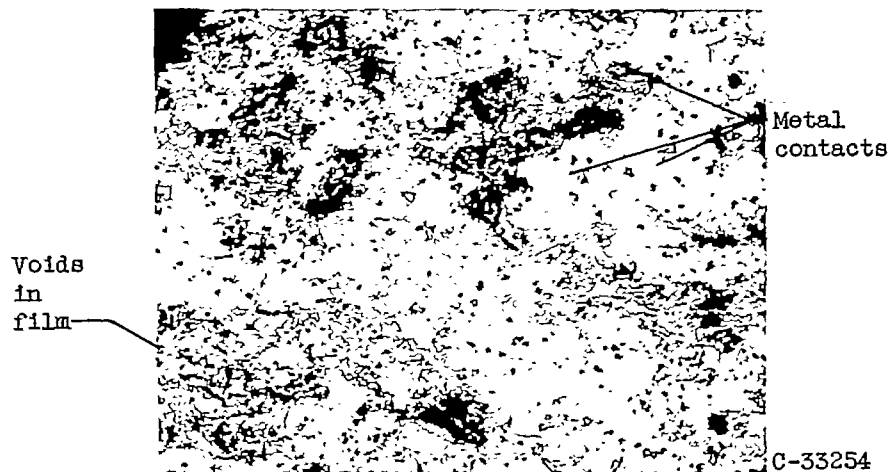
Disk

(c) Relative humidity, 65 percent.

Figure 6. - Continued. Photomicrographs showing surfaces of disk and slider after 6 hours sliding time at various humidities. Steel on steel lubricated with powdered MoS_2 . Magnification: disk, X100; slider, X50.



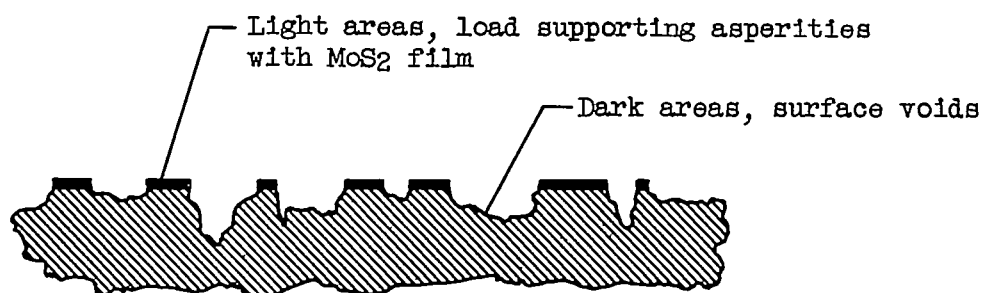
Node of slider



Disk

(d) Relative humidity, 86 percent

Figure 6. - Concluded. Photomicrographs showing surfaces of disk and slider after 6 hours sliding time at various humidities. Steel on steel lubricated with powdered MoS₂. Magnification: disk, X100; slider, X50.



Surface profile sketch

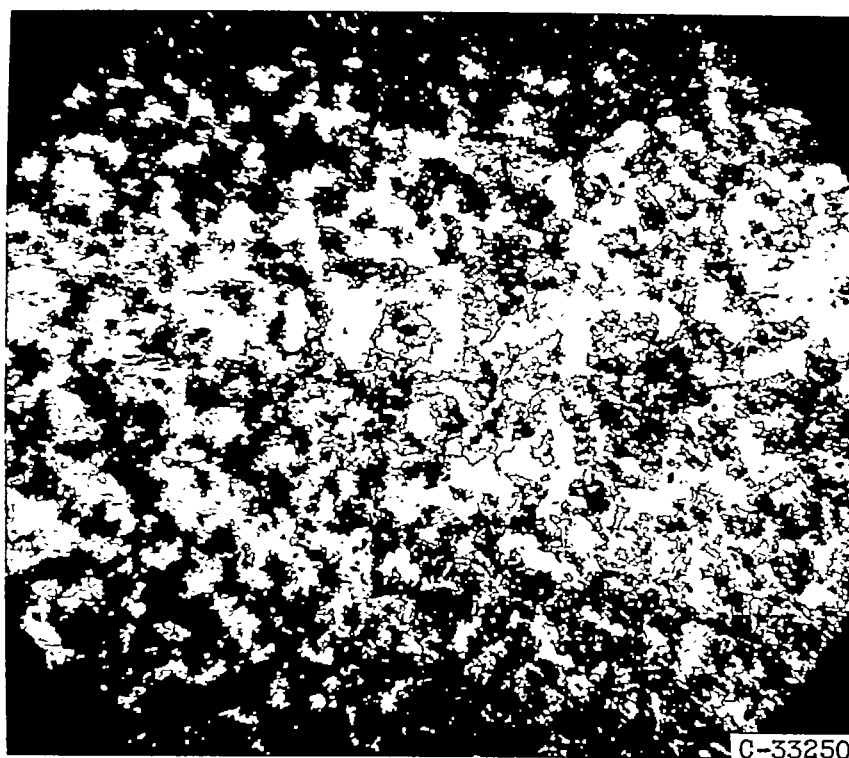


Figure 7. - Photomicrograph of surface of rubbed film showing MoS₂ shearing confined to tips of asperities (noncontinuous film).

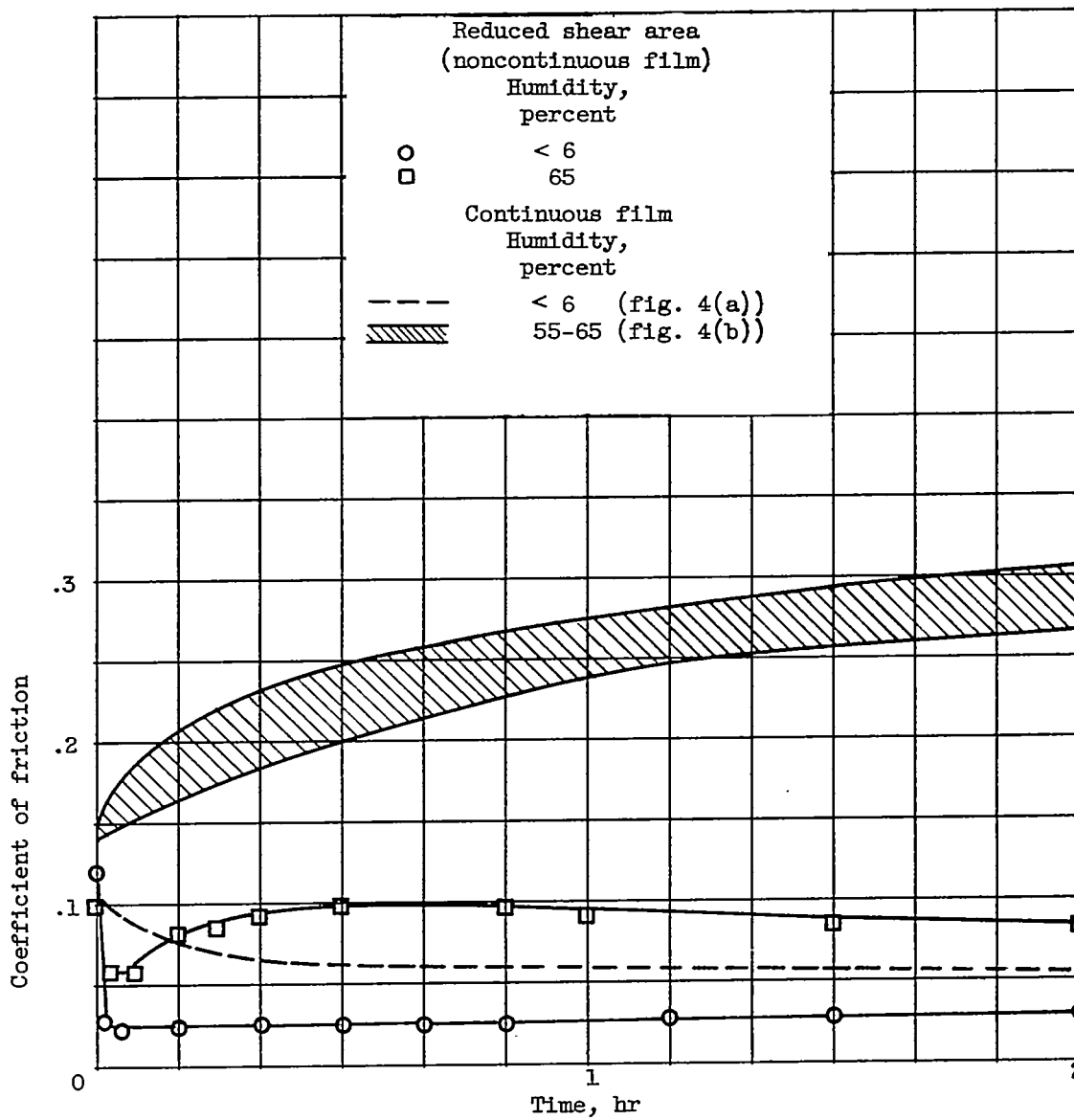


Figure 8. - Effect of reduced shear area on disk on friction coefficients for steel specimens lubricated with MoS_2 powder at high and low humidities. Reduced shear area obtained by using an insufficient amount of powder to form a continuous film. Load, 40 pounds; speed, 11 rpm (5.7 ft/min).

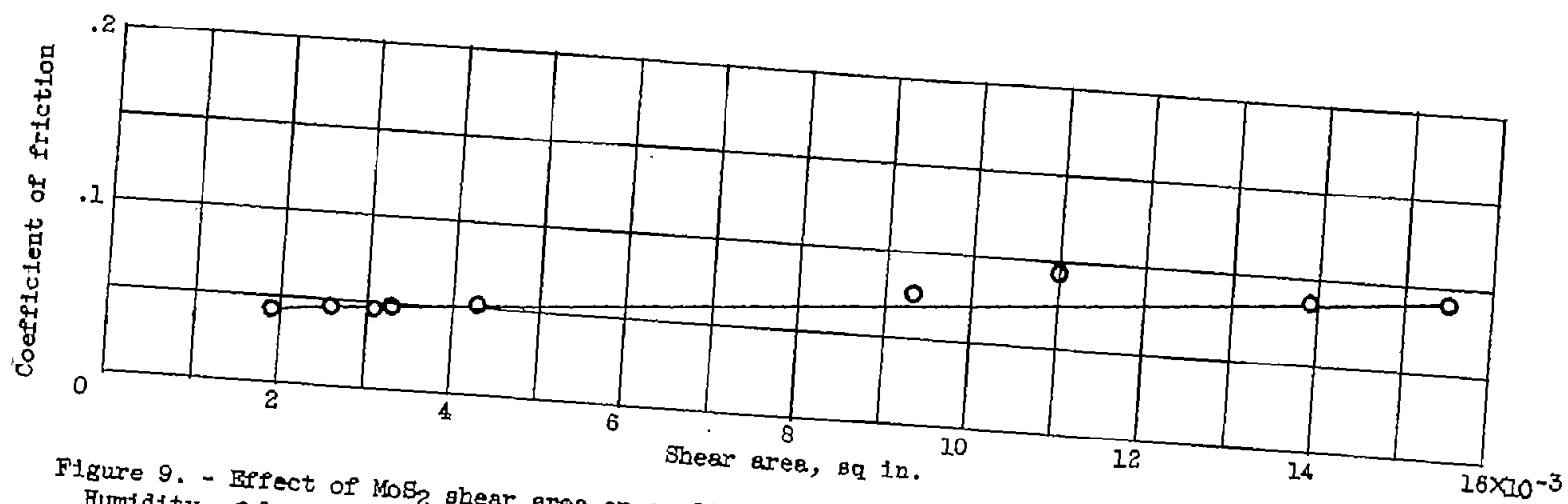


Figure 9. - Effect of MoS₂ shear area on coefficient of friction with powdered MoS₂ and steel specimens. Humidity, < 6 percent; load, 40 pounds; speed, 11 rpm (5.7 ft/min).

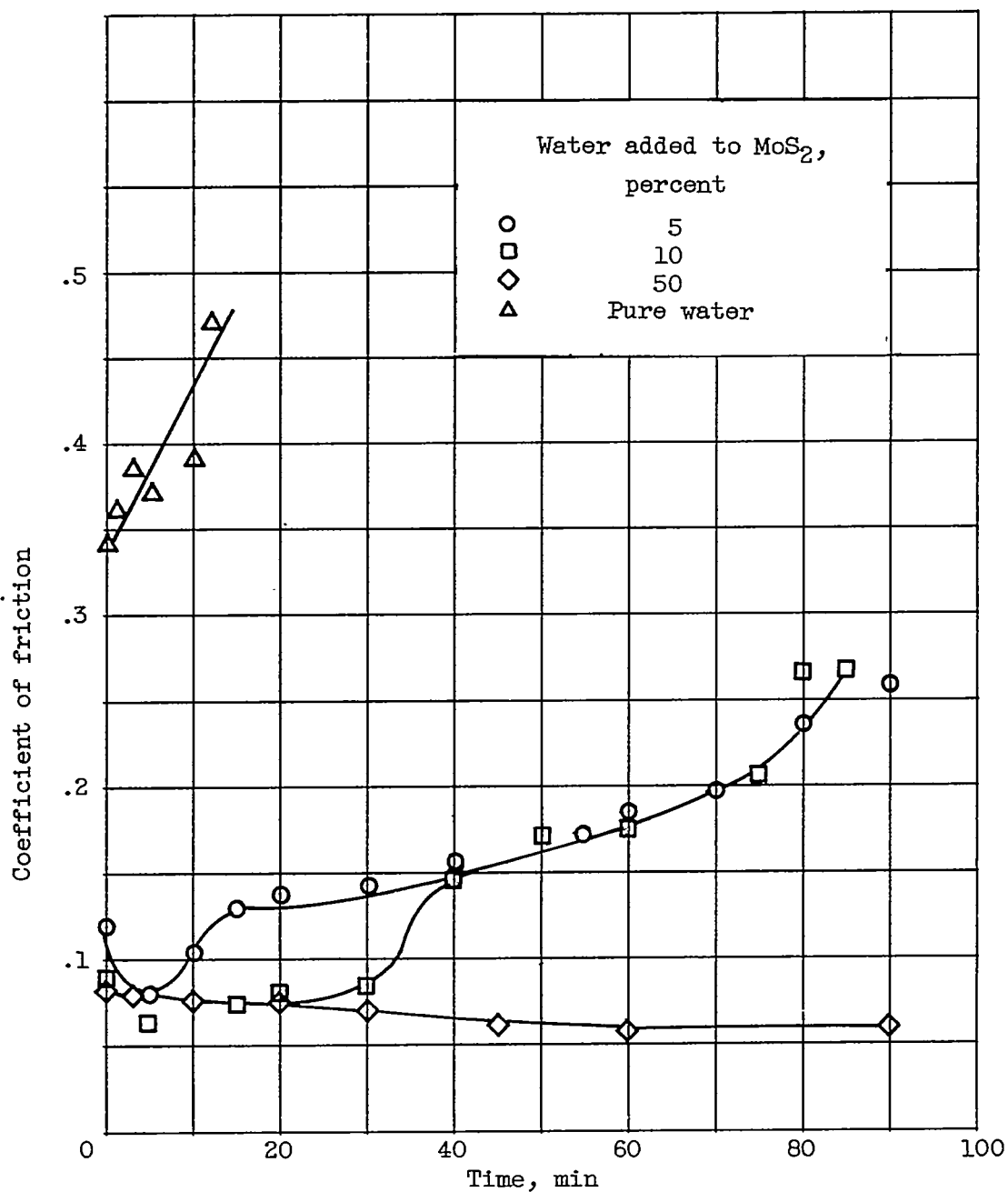


Figure 10. - Effect of additions of various amounts of water on friction coefficients of steel specimens lubricated with MoS₂, at room humidity. Load, 40 pounds; speed, 11 rpm (5.7 ft/min).

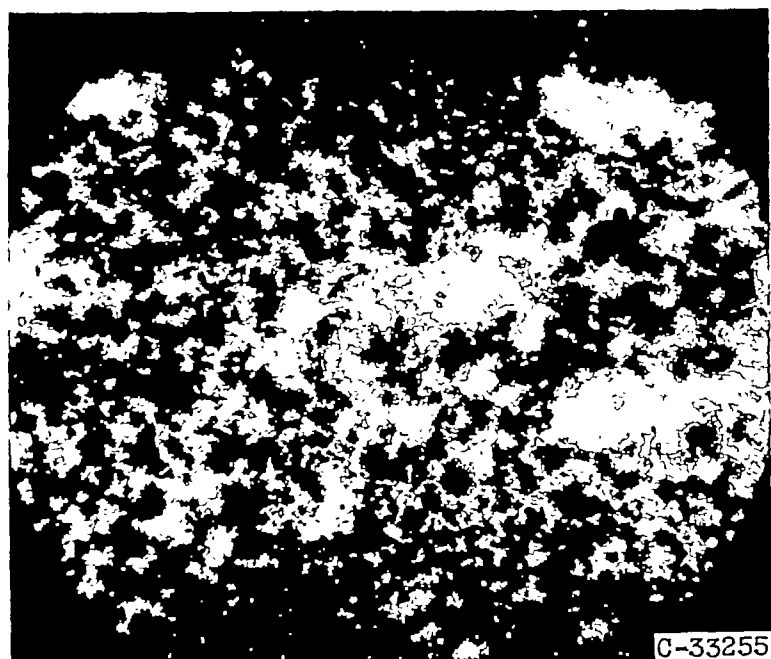


Figure 11. - Photomicrographs of surface of disk after 5 minutes sliding using MoS_2 plus 10 percent water as lubricant.

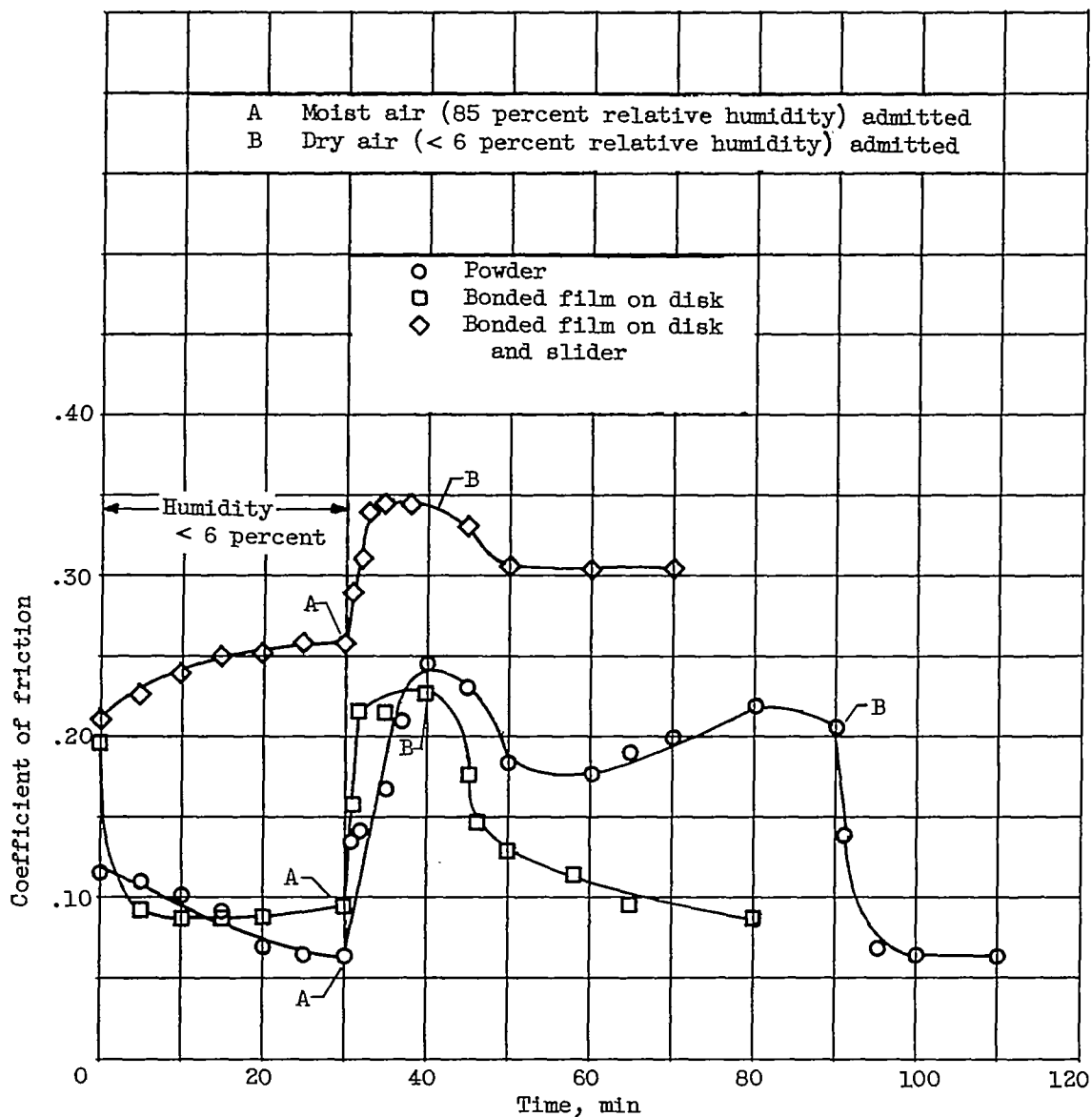


Figure 12. - Effect of admission of moist air on coefficients of friction for MoS_2 applied by various methods to steel specimens. Load, 40 pounds; speed, 11 rpm (5.7 ft/min).